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Determining the Complete Residual Stress Tensors in SOS Hetero-epitaxial Thin Film Systems by the Technique of X-Ray Diffraction

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Abstract. This paper investigates residual stress of epitaxial silicon film on SOS thin film systems. The emphasis was to develop a method to obtain accurately the complete residual stress tensors. It was found that using the multiple asymmetric X-ray diffraction method to measure strains in 13 [hkl] directions, the complete residual stress tensors can be determined reliably. The results were verified by both the Raman Backscattering and the substrate curvature methods.

Introduction

High residual stresses in hetero-epitaxial thin films of silicon on sapphire (SOS) systems are undesirable. Even when the stresses are well below the fracture strength of silicon, they can significantly affect the electronic transport properties and mobility of the material [1, 2]. Stress distributions and their generation mechanisms in a hetero-epitaxy SOS thin film system are complicated because of the single crystal anisotropy of the materials, their differences in thermal expansion properties and lattice mismatch.

Residual stresses in SOS wafers have been studied since 1960s, of which some are listed in Table 1. Dumin [3] measured the deformation of relatively thick films of SOS wafers using Zeiss light section microscope, and predicted the residual compressive stress based on the linear beam theory. Hughes [4] theoretically estimated the residual stress induced by differences of thermal expansion coefficients between silicon and sapphire materials, and correlated it with the piezoresistance-effects. Raman Scattering [5] is another method of estimating the residual stresses in SOS, which has been regarded as a powerful tool to characterize the average stress in as_grown and epi-fixed SOS wafers after ion-implantation and annealing [6,7,8]. X-Ray Diffraction (XRD) method [9] has also been utilized to measure strains for stress calculation.

Semiconductor thin films systems are mostly of a multilayered structure. Thus a tri-axial stress state is often expected. Therefore, the characterization of the magnitude and direction of principal stresses are crucial for the systems' reliability and their electrical mobility improvement. To have a successful residual stress characterization, the essential information required is an accurate measurement/prediction of a complete stress tensor at a point of interest in SOS wafer. However, all the aforementioned techniques are based on the assumption of equi-biaxial conditions or biaxial stresses, as highlighted in Table 1, which are unable to provide a complete residual stress tensor.

This paper aims to obtain a complete, accurate three-dimensional residual stress tensor in SOS thin film systems using the XRD technique.

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Table 1 Examples of residual stress investigations in as-grown SOS systems

Year	*Author*	*Technique*	*Assumptions*	*Thickness [μm]*	*Deposition Temperature*	*Film Stress [MPa]*
1965	D.J. Dumin[3]	Beam Curvature	Axial Stress	1-46	1100°C	-100~-1000
1972	A.J. Hughes[4]	Theoretical Estimations	Biaxial Stresses	1.5-1.8	1100°C	σ_{11}=-890 σ_{22}=-950
1979	TH. Englert[5]	Raman	Equi-Biaxial Stress	0.6-0.9	930°C	$\sigma_{11}=\sigma_{22}$= -700±30
1986	Thad Vrelland, Jr.[9]	X-Ray Diffraction	Biaxial Stress	0.4	1100°C	$\sigma_{[100]}$= -920±160 $\sigma_{[010]}$= -980±170
2005	Qi-Yuan Wang[6]	Raman	Equi-Biaxial Stress	0.5	980°C	-800
				0.2		-700

Residual Stress Characterization by X-Ray Diffraction Technique

The XRD stress analysis technique has been widely studied and standardized in polycrystalline materials [10], in which the diffractions are obtained from randomly oriented grains, with a series of inclinational angles Ψ. At the macroscopic scale, the lattice deformation of a crystalline material can be most directly measured by diffraction techniques. With controlled measurement directions, a complete strain tensor can be derived. However, in single crystal materials, the measuring directions are limited by the crystallographic orientations of (h k l) planes that can contribute to diffraction; thus 3-axes sample alignments are required to adjust the measured (hkl) plane in the most exact diffraction condition. This adds to the difficulties in measurement. Furthermore, the reliability of the calculated strain tensor could be degraded due to the limitation of the diffraction planes for analysis.

There are two main considerations when employing single crystal diffraction for residual stress analyses: the accuracy of the lattice spacing determined from peak shift and the value of stress-free lattice constant d_0. The conventional d-$\sin^2\Psi$ model can determine the stress from the slope of the d-$\sin^2\Psi$, which eliminates the error related to the stress free d_0. However, in order to determine a tri-axial stress state, it requires at least 3 sets of $\pm\psi$ at certain Φ azimuth angles, which is difficult to attain in single crystal structures; Multiple Regression [11, 12, 13] stress analysis model has been proposed with a plane-stress assumption, by χ-ψ axes oscillation method, which is equipped with a positional sensitive proportional detector; from which, the stress free lattice constants d_0 can be solved as an intercept of the multiple regression system. However, in order to determine the accurate tri-axial stress tensor, much more measuring directions are required to avoid the multicollinearity problem. Practically, this is not a feasible solution.

The authors found that the least square method [14] is the most appropriate to solve the single crystal tri-axial residual stress measurement issue. Accurate macro-strain tensors can be obtained by solving over-determined linear equations from multiple [hkl] asymmetric diffractions. From their work, the stress tensor accuracy was improved by more representative and arbitrarily distributed measuring [hkl] directions in 90° open Euler Cradle diffractometer (Table 2), in which, higher inclination angle up to 80° was achieved by psi offsets. Furthermore, in individual direction, the measured strain was improved by high angular resolution, along [hkl] with high diffraction angles. Therefore, higher accuracy was achieved in characterizing SOS residual stress tensors.

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Experiment

The samples investigated were 6 inch (001) Silicon on (10ī2) orientation Sapphire wafers, grown by CVD on a 600μm thick sapphire substrate*.

A residual strain (stress) tensor was calculated by combining the averaged strain in each measuring direction and assumed that strain distribution along the film thickness is uniform. The X-Ray diffraction measurements were performed on Philips Panalytical (MRD) diffractometer, equipped with a line focused high-resolution setting, in which the high intensive quasi-paralleled beam is collimated by an X-Ray Mirror, and a narrow band of diffraction angle from CuKα1 radiation is provided by four reflection Ge (220) monochromator with the resolution of ≤0.0059°. The Parallel Plate Collimator 0.18 ° was used to control the acceptance angle to detector. The results were made insensitive to the sample displacement.

To obtain the precise 2θ, it is necessary to approach the measuring (hkl) planes to the exact diffraction condition (diffraction maxima). This was done by optimizing the angle (Φ, ψ, ω) using consecutive scanning around the diffraction position in the Eulerian Cradle.

Determination of the Strain and Stress Tensors

As illustrated in Fig. 1, the strains ε_{11}, ε_{22}, ε_{33} in the sample coordinate are coincide with crystallographic directions [100], [010] and [001] in crystal coordinate, respectively. Although there are possible miscut and misorientation errors between the nominated and sample crystallographic directions, their effect is negligible if compared with the high residual stresses. $\varepsilon_{\Phi\Psi}$ is the strain in the measuring direction in the laboratory coordinate. The angles of Φ and Ψ are pre-determined azimuth and inclinational angles relative to [100] and [001] directions.

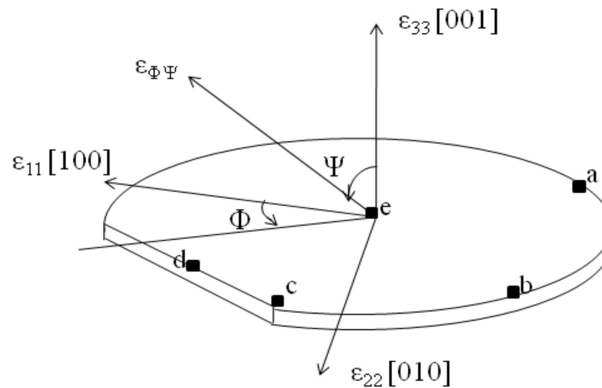

Fig. 1 Coordinate Settings

In an XRD stress analysis, an important step is to transform the measured strains in the laboratory coordinate $\varepsilon_{\Phi\Psi}$ to the 6 strain tensor components ε_{ij} (i, j =1, 2, 3) in the sample frame using [10].

$$\varepsilon_{\phi\psi} = (\cos^2\phi\sin^2\psi)\times\varepsilon_{11} + (\sin 2\phi\sin^2\psi)\times\varepsilon_{12} + (\cos\phi\sin 2\psi)\times\varepsilon_{13} + (\sin^2\phi\sin^2\psi)\times\varepsilon_{22} + (\sin\phi\sin 2\psi)\times\varepsilon_{23} + (1-\sin^2\psi)\times\varepsilon_{33} \tag{1}$$

* The processing conditions such as film thickness and film growth rate cannot be given here due to the nondisclosure policy of Sapphicon Semiconductor Pty Ltd.

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The measured $\varepsilon_{\Phi\Psi}$ and direction coefficients represented by cosΦ etc are known input data in Eq. (1). When sufficient strains, $\varepsilon_{\Phi\Psi}$, in different [hkl] directions are obtained, ε_{11}, ε_{22}, ε_{33} can be determined. Normally, to minimise the errors induced in the measurement aforementioned, it is desirable to obtain an over-determined set of linear equations of Eq. (1) and solve for ε_{11}, ε_{22}, ε_{33} by the least square method. Table 2 lists the strains measured in 13 [hkl] directions, represented by Φ, Ψ angles, where $\varepsilon_{\Phi\psi}$ is the experimental measured strain, and $\varepsilon`_{\Phi\psi}$ is the strain calculated from the transformed strain tensor. The table shows that the difference between $\varepsilon_{\Phi\psi}$ and $\varepsilon`_{\Phi\psi}$, Δ, is very small and is within 0.01% in each [hkl] direction, indicating that the strain tensor obtained was reliable.

Table 2 XRD measured and calculated strains in 13 [hkl] directions.

[hkl]	*Φ(°)*	*ψ(°)*	$\varepsilon_{\Phi\psi}$ *(Measured)*	$\varepsilon`_{\Phi\psi}$ *(Predicted)*	$\Delta = \varepsilon`_{\Phi\psi} - \varepsilon_{\Phi\psi}$
004	45	0	0.3031%	0.3050%	0.00185%
115	45	15.79	0.2450%	0.2480%	0.0030%
206	0	18.44	0.2352%	0.2367%	0.0015%
315	18.44	32.31	0.1033%	0.1044%	0.0011%
335	45	40.32	0.0090%	0.0040%	-0.0050%
404	0	45	-0.0393%	-0.0419%	-0.0026%
444	45	54.736	-0.1698%	-0.1676%	0.0022%
-153	168.69	59.53	-0.2341%	-0.2247%	0.0094%
353	59.04	62.77	-0.2602%	-0.2596%	0.0006%
-351	149.04	80.27	-0.3931%	-0.3953%	-0.0021%
20-6	0	-18.44	0.2374%	0.2333%	-0.0040%
31-5	18.44	-32.31	0.1061%	0.1098%	0.0037%
40-4	0	-45	-0.0380%	-0.0476%	-0.0096%

With the residual strain tensor obtained above, the complete residual stress tensor could be calculated using the Hooke's Law with the elastic constants of silicon [15]. The following are the strain and stress tensors for the wafer with a Si film. It can be seen that the stresses are basically a biaxial state within the plane of the film.

$$\begin{bmatrix} \varepsilon_{11} & \varepsilon_{12} & \varepsilon_{13} \\ \varepsilon_{21} & \varepsilon_{22} & \varepsilon_{23} \\ \varepsilon_{31} & \varepsilon_{32} & \varepsilon_{33} \end{bmatrix} = \begin{bmatrix} -0.394\% & 0.014\% & 0.003\% \\ 0.014\% & -0.410\% & -0.018\% \\ 0.003\% & -0.018\% & 0.305\% \end{bmatrix} \quad \begin{bmatrix} \sigma_{11} & \sigma_{12} & \sigma_{13} \\ \sigma_{21} & \sigma_{22} & \sigma_{23} \\ \sigma_{31} & \sigma_{32} & \sigma_{33} \end{bmatrix} = \begin{bmatrix} -723.32 & 23.00 & 4.51 \\ 23.00 & -741.41 & -28.54 \\ 4.51 & -28.54 & -9.96 \end{bmatrix}$$

The principal stresses could be determined from the stress tensor above [16] as $\sigma_1 = -8.8$MPa, $\sigma_2 = -707.9$MPa and $\sigma_3 = -758$MPa. With the plane stress assumption, the principal stress direction was found to be 34.3 degrees from the [100] axis. The von Mises stress [16] in this case was 725.4MPa. Although this stress magnitude is much smaller than the silicon failure stress (7GPa), it is large enough to be considered when the electrical performance of the film is critical concern [1,4].

Verification

To verify the XRD measured residual stresses, some other techniques were used to measure some residual stress components although these techniques cannot provide a complete stress tensor and they are based on some strong assumptions.

Raman Spectroscopy Method. The Raman experiment was conducted with the backscattering configuration [5]. The Raman backscattering stress analysis technique is based on the assumption of equi-axial plane stress state, and the residual stress is calculated by the shift of LO phonon frequency $\Delta\omega$ (cm^{-1}), i.e., $\sigma = -249 \times \Delta\omega$ (MPa) in the case of silicon.

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Five points (from a to e in Fig. 1) were measured in the as-grown SOS wafer. The reference silicon peak position was $\omega_0 = 520.4$ cm^{-1}. From the measured Raman shifts $\Delta\omega$ at those points, the average residual stress obtained was -703.84MPa, which is the average stress of the five points ($\sigma_a = -706.5$MPa, $\sigma_b = -683.3$MPa, $\sigma_c = 700$MPa, $\sigma_d = -720.7$MPa, $\sigma_e = -708.7$MPa). The Raman result is very close to the XRD measured value (-732.95MPa, the average of the principal stresses in the plane σ_2 and σ_3).

Substrate Curvature Method. With the high resolution configuration, the wafer curvature caused by residual stresses could be determined by the X-ray rocking curve method [17], which measures the rocking curve ω of the substrate peak for two locations of distance *S* (Fig. 2) to give rise to the radius of curvature *R*. In SOS wafer, *ω* was measured at 6 points throughout the surface, and an average *R* was determined to be 152m.

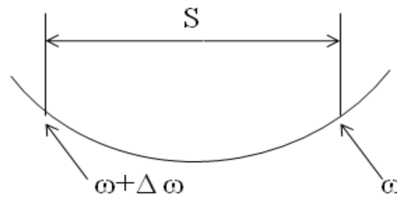

Fig. 2 X-Ray Rocking Curve curvature method

Then with the assumption of equi-biaxial stresses, the residual stress in the silicon film could be obtained by using the well-known Stoney Equation [18] when the elastic constants of sapphire in Table 3 were used, as listed in Table 4. The average was found to be -717.82MPa which again is very close to the XRD measured value of -732.95MPa.

Table 3 Sapphire Elastic Constants

[hkl]	*[001]*	*[100]*	*[010]*
E(GPa)	386	381.88	381.88
ν	0.25	0.25	0.25

Table 4 Residual stress by curvatures method

	R(m)	*Stress(MPa)*
Minimum	145.37	-750.56
Maximum	158.72	-687.43
Average	152	-717.82

Thermal Expansion Method. The residual stresses in the silicon film can also be theoretically calculated by considering the difference in thermal expansion coefficients between Si (α_0=3.9x10^{-6}/°C) and Al_2O_3 (α_1=8.31x10^{-6}/°C and α_2=9.03 x10^{-6}/°C) where α_1 and α_2 are the coefficients in directions perpendicular and parallel to the c-axis of Al_2O_3 respectively [4]. This method brought about $\sigma_{11} = -714.75$MPa and $\sigma_{22} = -760.57$MPa, which is very consistent with the $\sigma_{11} = -723.32$MPa and $\sigma_{22} = -741.41$MPa determined by XRD technique presented above.

Conclusions

1. A complete stress tensor in an SOS thin film system was obtained by the XRD technique. The accuracy of the conventional least square XRD method was improved by multiple [hkl] assymetric diffractions in 13 representative diffraction directions and higher anguler resolution for individual strains. By combining ω and ψ offsets, more arbitrary inclination angles scattered from 0-80° was achieved by using the 90° Open Euler Cradle 4-Circle diffractometers.
2. The reliability of the results was verified by three different techniques, ie, the Raman spectroscopy method, the substrate curvature method and the thermal expansion method.
3. For the thin film system analysed, although the compressive residual stress is much smaller than the critical frature stress of silicon, it is big enough to influence the electrical properties of an SOS device.

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